

Appl. No. : **10/530,781**
Filed : **April 7, 2005**

SUMMARY OF INTERVIEW

Exhibits and/or Demonstrations

none

Identification of Claims Discussed

none

Identification of Prior Art Discussed

none

Proposed Amendments

none

Principal Arguments and Other Matters

Examiner Christensen was contacted regarding the drawing objection. The difficulty of adding a Figure to respond to the drawing objection without addition of new matter was discussed. The Examiner consulted with his Supervisor and advised that he was willing to withdraw the drawing objection. Upon further consideration, the USPTO considered that the claims recite properties which may be difficult to represent in a drawing. Alternatively, the Examiner advised that we could submit a simple block drawing.

Results of Interview

The undersigned elected withdrawal of the drawing objection. Examiner Christensen agreed to withdraw the objection.

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REMARKS

Claim 6 has been cancelled. Claims 1, 2, 8, and 15 have been amended. Claims 1-5 and 7-21 are now pending in this application. Support for the amendments is found in the existing claims and the specification as discussed below. Accordingly, the amendments do not constitute the addition of new matter. Applicant respectfully requests the entry of the amendments and reconsideration of the application in view of the amendments and the following remarks.

Telephonic interview

Applicants thank Examiner Christensen for the helpful telephonic interview July 19, 2006 between Examiner Christensen and Che Chereskin. The Drawing objection was discussed. The Examiner's reconsideration of the Drawing objection is greatly appreciated.

Drawings

Applicants gratefully acknowledge withdrawal of the drawing objection as indicated in the Interview Summary from the Examiner mailed July 25, 2006 and also summarized herein (page 6).

Specification

The specification has been amended to correct a typographical error which was kindly pointed out by the Examiner. On page 3 of the specification, "200 mm" has been corrected to "20 mm". Support for the amendment is found, for example, in claim 8. Withdrawal of the objection is respectfully requested.

Claim objections

Claims 2, 8, and 15 have been amended to correct typographical errors and minor informalities as pointed out by the Office Action in items 5-8.

Withdrawal of the objections is respectfully requested.

Rejection under 35 U.S.C. § 102(b)

Claims 1-3 and 5 are rejected under 35 U.S.C. § 102(b) as being anticipated by Britto, et al. ("High Density Polyethylene Fraction with Supercritical Propane").

Britto et al. disclose a method for fractionating polymers using supercritical fluid fractionation (SFF) and CITREF. Britto discloses the use of an extraction column comprising a *stainless steel knitted mesh* as packing (see page 554, right column, last paragraph, and page 555, left column, second paragraph).

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Amended claim 1 is directed to a column wherein the packing of said column comprises elastic wires having a length per diameter (L/D) of at least 3. Basis for the amendment can be found in original claim 6 (now cancelled).

It is respectfully submitted that the column as presently claimed is not disclosed in Britto et al., since Britto et al. do not provide any indication on the L/D ratio of the mesh/wires present in the column.

Accordingly, it is respectfully submitted that Britto et al. do not teach all of the elements of claim 1 as amended.

It is further submitted that claims 2-3 and 5 depend from claim 1. Accordingly, Britto, et al do not teach all of the elements of claims 2-3 and 5, at least for the reasons presented above for claim 1.

In view of Applicants' amendments and arguments, reconsideration and withdrawal of the above ground of rejection is respectfully requested.

Rejection under 35 U.S.C. § 103(a) (Britto in view of Hazlitt)

Claims 7-18 are rejected under 35 U.S.C. § 103 (a) as being unpatentable over Britto, et al. ("High Density Polyethylene Fraction with Supercritical Propane") in view of US Patent No. 4,798,081 to Hazlitt, et al.

Britto et al. disclose a preparative TREF method for fractionating polymers using an extraction column comprising a stainless steel knitted mesh as packing.

Hazlitt et al. disclose a method and apparatus wherein a combination of high temperature continuous viscometry (HTCV) and ATREF techniques are used for the analysis of crystalline and semi-crystalline polymers and to determine crystallinity versus weight percent profile concurrently with viscosity average molecular weight. The columns used for performing the ATREF analyses in Hazlitt comprise small or large columns made of stainless steel, which are packed with cylindrical steel shots.

It is respectfully submitted that Britto, et al. and Hazlitt et al. both disclose a column, a device and a method which, taken together or separately, are different from the column, device and method claimed in the present invention.

Column

A difference between the columns as described in either Britto or Hazlitt and the present invention is the use of a different type of packing in the fractionation column. As set forth in present claim 1, a column with high density packing of "elastic wires having a length per diameter (L/D) of at least 3" is used, while the packing in Britto consists of a packed mesh and in Hazlitt of a column of cylindrical steel shots. Neither Britto nor Hazlitt teach or suggest a column packing containing wires having the indicated L/D ratio of at least 3.

The unexpected benefits provided by a packing comprising elastic wires having the indicated L/D ratio for the ATREF column according to the present invention are as follows:

- The fibers having the above indicated L/D ratio give a much higher surface for polymer separation and allow even very similar polypropylene samples to be distinguished as shown in the present patent application (see Example 2, pages 15-16 referring to Figures 4A, 4B, 5A and 5B).
- The wires provide a high surface area: this high specific surface area permits "controlled" crystallization of the polymer in the column when the temperature is decreased - this surface serves as "nucleating" agent- and its "controlled" re-dissolution when the temperature is raised. This leads to improved sensitivity, since no polymer is lost to the mobile phase, and to better baseline stability. The baseline stability obtained with the present column (see Example 1, pages 14-15 and particularly Figures 2A & B of the present specification) cannot be obtained with a column such as the one of Britto or Hazlitt.
- The elastic wires of the presently claimed invention provide a homogeneous packing without holes (*cfr*: a mesh contains holes of Britto et al.). This is important since the polymer has to precipitate from solution. If it remains suspended in the solvent (the mobile phase), it will be lost for the analysis and give baseline problems.
- The use of elastic wires as column packing instead of small metal particles, shots or plates (see for instance Hazlitt et al.) provides excellent thermal conductivity to the column. The elastic wires inside the column compensate for the pressure variation during the elution process. The packing with the elastic wires improves the baseline stability and consequently allows the use of a sensitive DRI detector during ATREF. When using small objects (glass, bronze, metallic shots, etc. – see for instance Hazlitt et al.)

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instead of wires, the free internal volume is fixed if one wants to obtain a homogeneous filling. Also the specific surface area will be lower at a same volume fraction due to less effective length/diameter ratio.

- The ATREF column packing has to remain homogeneous for high variations of temperature and pressure which occur during the analysis; This is achieved by the use of elastic wires according to the present claimed invention.

Neither Britto nor Hazlitt teach elastic wires according to the claimed invention. By incorporation of elastic wires into the column packing, the advantages listed above are achieved. These advantages were neither taught nor suggested by the cited documents.

Device

Regarding the device of claims 9-10, the device includes the column discussed above. Accordingly, the device is patentable for the reasons given above with respect to the column.

ATREF Method

Regarding the method claims, the method disclosed in Britto pertains to preparative TREF, not an ATREF method as applied in the presently claimed invention.

Regarding Hazlitt, the method in the present invention differs from the method in Hazlitt in that the presently claimed method recites the use of a different type of column and column packing (the column of claim 1). These differences are discussed above.

In addition, the present method differs from the method in Hazlitt in that in the present method polymer sample solution is crystallized in the column "while *keeping the solvent flowing through the column*" (see claim 11, emphasis added). In contrast, in Hazlitt solvent flow is stopped during the crystallization step. The pump is stopped during the crystallization step as clearly indicated in column 10, lines 65-68 of Hazlitt which teaches that: "the other end of the column is plugged". Consequently, the column is completely disconnected from the solvent flux. Maintaining the solvent flow through the column during the crystallization step is non-obvious for analytical TREF and provides unexpected benefits when using a DRI detector since the baseline remains always excellent, addressing the technical problem stated in column 12, lines 16-36 of Hazlitt. Hazlitt, et al. disclose that "an intense positive pen deflection on the chart recorder occurs due to DRI response, lasting as much as 1.5 hours [30 to 45 minutes], before a

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baseline condition is reestablished". Applicants' claimed method addresses this technical problem. By the practice of the method as claimed, a stable baseline is obtained (compare Figures 2A and 5A to Figures 2B and 5B of the present specification). Accordingly, claim 11 (and claims dependent thereon) are additionally patentable for the reasons stated above.

In addition, referring to point 25 of the office action, it is respectfully submitted that the amount of polymer sample added to the column in Hazlitt is not 0.5mg but 0.5 gm (thus 500 mg) which is 250 times greater than 2 mg, the polymer quantity that can be used in the present method. Much lower amounts of polymer sample need thus to be added for analyses according to the present invention (see Example 1 of the present specification, particularly page 15, lines 13-15). Application of an ATREF method producing an accurate TREF chromatogram with such a small quantity of polymer has not been previously disclosed nor suggested. Accordingly, claim 14, while being patentable for the reasons given above, is also patentable as the art does not teach or suggest this limitation.

Furthermore, regarding claim 19, the cooling rate applied in Hazlitt is lower (0.17°C/min) (see column 11, line 1-2) than in the present invention. Accordingly, the cited art does not teach or suggest the limitation as recited in claim 19.

versus Britto

The claimed column, device and method are patentable over Britto, et al. because Britto, et al. do not pertain to an ATREF method but to a TREF method and provide a different column and column packing. In addition, it is not possible to use the present column design for preparative TREF (as disclosed by Britto et al.) and *vice versa*, to use the column of Britto in the present ATREF method. Hence the two columns (the column as presently claimed and the column disclosed by Britto) are very different.

Britto et al. describes a column used for preparative TREF, which is not suitable for analytical TREF. It is noted that the use of a stainless steel mesh for the preparative TREF has been reported in the art (See for instance Viville et al., enclosed as Attachment A). Preparative TREF is a method which is designed to produce large quantities of polymer fractions from the original sample, typically, several grams. Therefore a large quantity of initial sample has to be charged in the column (about 10 g of HDPE in Britto et al.). This high quantity gives a high

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polymer concentration in the column and produces a high viscosity of the solution. This is clearly stated in Britto, et al. that the HDPE pellets are "charged to the extraction column, heated and pressurized to permit complete polymer dissolution, and allowed under buoyancy mixing for 6h to ensure uniform concentration in the vessel" (see page 555, , column 2, first full paragraph). In order to realize this mixing as well as the subsequent elution steps when the temperature is increased stepwise, it is necessary to have a large free volume in column. For this reason, the "mesh" actually has large holes between the wires, in which the highly viscous polymer solution can freely circulate. Such a design is suitable for preparative TREF even if it does not entirely guarantee the column will not be blocked for high molecular weight polyethylene samples. If the present column with highly compressed wires (fibers) was to be used in a preparative TREF method as disclosed by Britto et al., the free internal volume would be much too small, and immediate blocking of the column would occur and no analysis could be performed.

Compared to preparative TREF, the scope of the present ATREF method is to monitor the eluting fractions from the column, and therefore to obtain a chromatogram which details the sample microstructure, e.g. branching in the case of polyethylene, or tacticity in the case of polypropylene). In this case, the injected polymer quantity is several orders of magnitude less than for preparative TREF (typically milligrams) and the column must have a higher resolution power than for preparative TREF. The low injected polymer concentration in the column implies a low viscosity of the polymer solution, which allows the use of compressed fibers (small free volume). Very importantly, the fibers having the above indicated L/D ratio give a much higher surface for polymer separation and allow to distinguish even very similar polypropylene samples as shown in the present patent application (see Figure 5B, for example). The mesh described in Britto et al. does not provide the resolution power to analyze the polymer sample.

versus Hazlitt

It is noted that although Hazlitt describes the problem of baseline stability (see column 12, line 16-26), no indication is given that such problems may be resolved by a specific choice of the column packing and physical properties (length/diameter ratio) of elastic wires used in the column packing. There is no teaching or suggestion in Hazlitt, et al that would motivate one of

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ordinary skill in the art to amend the column packing in order to arrive at the present column packing and the present column.

Additionally, as noted above, Hazlitt does not teach or suggest a method for performing ATREF wherein polymer sample solution is crystallized in the column while *keeping the solvent flowing* through the column.

versus Hazlitt and Britto

In contrast to what is stated in point 22 of the office action, even upon introduction of the column of Britto in the method of Hazlitt, one would not arrive at the present invention, since Britto et al. disclose a different column that is suitable for another type of analysis (TREF) but not suitable for ATREF analysis. The combination of Britto, et al. and Hazlitt, et al. would not lead one of ordinary skill in the art to the invention claimed. Furthermore, the teachings of Britto et al. and Hazlitt, et al. are different from the column and method of the present invention, as indicated above.

In view of the above, it is submitted that use of the present column and device in ATREF methods is not obvious in view of the teachings of Britto and Hazlitt, either taken alone or in combination. Withdrawal of the rejection is respectfully requested.

Rejection under 35 U.S.C. § 103(a) (Britton in view of Halasz)

Claims 4 and 6 are rejected under 35 U.S.C. § 103(a) as being unpatentable over Britto, et al. ("High Density Polyethylene Fraction with Supercritical Propane") in view of US Patent No. 3,340,085 to Halasz, et al.

The limitations of claim 6 are now incorporated into claim 1. Accordingly, this ground of rejection will be considered as it applied to claim 1 as amended.

Halasz et al. disclose packed columns for use in gas chromatography and a packing for a gas chromatographic column. The packing of Halasz is formed by an inert core which can be many things including small stainless steel mesh rings, which are covered by a porous layer. Halasz, et al differ from the present invention in disclosing a porous layer that covers the inert core. In the presently claimed invention, the elastic wires are not covered by any porous layer.

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Furthermore, Halasz discloses a packing for gas chromatography and does not provide any motivation to use the disclosed packing in an ATREF column. The packing material of Halasz is specifically adapted to gas chromatography. As stated by Halasz, "said column [contains] a packing of gas-impervious bodies carrying porous layers on their surfaces" (col. 1, lines 61-62). The purpose of the porous material is not only support, but also to provide an absorbent material within which the separation process proceeds, at least partially (see col. 1, line 65-col. 2, line 2). Halasz does not provide any hint about the fact that elastic wires will improve the baseline in analytical TREF. In fact, Halasz teaches that "the material of which these cores are made is of no greater importance, as long as it is gas-impervious and inert. Glass or bronze serves well" (col. 4, lines 13-15). Halasz also teaches that the shape is not an important feature. "Instead of spheres, cores of another shape can also be be [sic] used. As an alternative, cores of a shape common in distillery technique, such as Raschig rings, Berl saddles, Steadman packings wire mesh rings and wire helices, etc. are operable" (col. 4, lines 26-30). In contrast to the teaching of Halasz, the present claims specify that "said column comprises elastic wires having a length per diameter (L/D) of at least 3" claim 1). Applicant attests that the glass beads, thin bronze beads and metal-coated glass beads (see claim 2 of Halasz) are not different (from a geometrical standpoint) from cylindrical steel shots used by Hazlitt and will give the same baseline problem when using the DRI detector. Halasz does not teach ATREF and does not teach the importance of using elastic wires having a length per diameter (L/D) of at least 3 as claimed. Accordingly, there is nothing in Halasz that would lead one of ordinary skill in the art to use elastic wires as claimed.

Furthermore, the cited references do not provide motivation apply the column provided in Halasz, in the system of Britto, because the two documents pertain to another two different types of analysis (TREF versus chromatography).

Even if one combines the teachings of Britto and Halasz, one of ordinary skill in the art would not be able to arrive at the present invention. As discussed above, Britto teach the TREF method and do not teach maintaining a flow of solvent during crystallization as in the present claims. Halasz teaches that the packing material can be made of any inert material and can be of virtually any shape. Accordingly, Halasz provides insufficient guidance to one of ordinary skill

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in the art to use the specific elastic wires as claimed. Halasz emphasizes the importance of a coating, which is not a part of the presently claimed invention.

In view of Applicants' amendments and arguments, reconsideration and withdrawal of the above ground of rejection is respectfully requested.

Rejection under 35 U.S.C. § 103(a) (Britto in view of Hazlitt in view of Minami)

The Examiner has rejected claims 19 and 21 as being unpatentable over the combination as applied to claim 11 (Britto in view of Hazlitt), further in view of US Patent No .6,770,355 to Minami, et al. The Examiner asserts that it would have been obvious to one of ordinary skill in the art to modify the system taught by Hazlitt, et al. by using heating and cooling rates greater than 0.5 °C as disclosed by Minami, et al because ATREF generally uses smaller sample sizes and it would be within the skill on one in the art to determine appropriate heating and cooling rates depending on the polymer sample and to choose the quickest feasible rate in order to obtain results more quickly. However, since claims 19 and 21 depends from claim 11 (which depends from claim 1), which is neither taught nor suggested by Britto in view of Hazlitt, the invention defined in claims 19 and 21 is also patentably distinguished from the references, alone or in combination. Applicants respectfully request the withdrawal of the rejection.

CONCLUSION

In view of Applicants' amendments to the claims and the foregoing Remarks, it is respectfully submitted that the present application is in condition for allowance. Should the Examiner have any remaining concerns which might prevent the prompt allowance of the application, the Examiner is respectfully invited to contact the undersigned at the telephone number appearing below.

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Please charge any additional fees, including any fees for additional extension of time, or credit overpayment to Deposit Account No. 11-1410.

Respectfully submitted,

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